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Technical Report No. 32-168

Volume Changes and Poisson's Ratio of Polyurethane Propellants Under Tensile Deformations

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VOLUME CHANGES AND POISSON'S RATIO OF POLYURETHANE PROPELLANTS UNDER TENSILE DEFORMATIONS

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ABSTRACT

Volume changes accompanying tensile strain have been measured by hydrostatic weighing techniques. The inherent error of the method is analyzed and the limitations of the apparatus and the accuracy of the results are discussed. Four polyurethane propellants have been studied at 25°C, using strains from 2.5% to break, in 2.5% increments. The volume changes are interpreted in terms of a change in Poisson's ratio. When the latter is defined in terms of the Cauchy strains, Poisson's ratio decreases at a greater rate than calculated for an incompressible material. The region of 2.5% strain is masked by experimental error, but the results indicate volume changes at all strains.

INTRODUCTION

With the expansion of composite solid propellants into large rocket motor applications, the development of reliable design and fabrication methods has become a problem of increasing concern. In order to insure adequate structural integrity of solid propellant rocket motors, it becomes necessary to predict the behavior of a complex grain in a multi-axial stress state from simple laboratory tests, usually carried out under uniaxial loading. As a consequence, an increasing emphasis is being placed on approximate mathematical analyses of stresses and strains in solid propellant rocket motors; these are generally based on simplified models and assumed boundary conditions.

Virtually all strain analyses require a knowledge of the Poisson's ratio ν. This parameter assumes a special physical significance when applied to composite solid propellants. It is directly related to the increase in volume of a propellant specimen, when subjected to a tensile test, resulting from the separation of the binder from the oxidizer and the formation

of vacuoles. Previous work at the Jet Propulsion Laboratory (1) has shown that vacuole formation occurs before the maximum value of stress (usually called the tensile strength σ_m) is reached, and it was believed that it began rather abruptly at some critical value of strain ϵ_c which is roughly equal to 2/3 of the strain at maximum stress, ϵ_m. However, a much more accurate method for determining volume changes was needed. On the other hand other studies, notably at Aerojet (2), where sample dimensions were determined photographically during straining, have indicated that ν changes continuously during straining. However, the experimental error in these data prevented a definitive conclusion.

CALCULATIONS OF POISSON'S RATIO

Classical strain analyses define Poisson's ratio in terms of pure homogeneous Cauchy strain as

$$\nu = - \frac{\epsilon_w}{\epsilon_1} \tag{1}$$

where ϵ_1 is the longitudinal strain in the direction of the applied stress and ϵ_w is the lateral strain. In small-deformation elastic theory, it is approximately true that ν is constant and equal to 1/2 if the material is incompressible; it is exactly 1/2 only for infinitesimal strains. For finite strains, however, such as occur in propellant grains, this is no longer true, and it can be easily shown that Poisson's ratio for an isotropic, incompressible material is a nonlinear function of the tensile strain given by

$$\nu = \frac{1}{\epsilon_1} \left[1 - (1 + \epsilon_1)^{-1/2} \right] \tag{2}$$

If a volume change occurs on straining, it is convenient to express Poisson's ratio as a function of the principal strain ϵ_1 and the volume ratio V/V_0. In order to do this, it is still necessary to assume that the material is isotropic and that the volume change is given by the relation

$$V/V_0 = (1 + \epsilon_1)(1 + \epsilon_w)^2 \tag{3}$$

Solving Eq. (3) for ϵ_w and substituting in Eq. (1), the expression for Poisson's ratio becomes

$$\nu = \frac{1}{\epsilon_1} \left[1 - \left(\frac{V}{V_0} \right)^{1/2} (1 + \epsilon_1)^{-1/2} \right] \tag{4}$$

For an incompressible material $V/V_0 = 1$ and then Eq. (4), of course, reduces to Eq. (2). In this work, Eq. (4) will always be used to calculate Poisson's ratio for propellants unless indicated otherwise.

Unfortunately, other definitions of Poisson's ratio are sometimes used for convenience in reducing and interpreting experimental results (see for example Ref. 3). Any definition used with Eq. (3) will yield expressions analogous to Eq. (2) and (4), but the results are numerically quite different. This is seen from equations summarized in Table I. In addition, the same three functions for incompressible materials are compared in Fig. 1, showing vastly divergent results. This point is emphasized because numerical values obtained by one definition are sometimes quoted and used in strain analyses based on a different definition of ν. Obviously, such a procedure makes any conclusions quite meaningless. Since ν_1 seems to be the definition most commonly employed in stress analysis, it will be used here. Values read from the $\nu_1 - \epsilon$ plots could then be employed to calculate ν_2 or ν_3 at a later date if desired.

EXPERIMENTAL

The volume changes which occur during straining of highly filled systems can be measured with the best precision by a dilatometric technique. Such a study was carried out with glass bead-polyvinyl chloride composites (3), but the work was not extended to actual propellant formulations. The dilatometer used in the previous study had some disadvantages and required samples in the form of elongated rings; since propellant specimens could not be readily prepared in this shape, a new apparatus was devised. Rainbird and Vernon (4) have employed a hydrometer technique, using samples in the form of dumbbells for tensile tests and cylinders for compressive tests.

A new device based on the same principle was independently developed at JPL. The apparatus, shown in Fig. 2, determines the volume of propellant specimens gravimetrically by measuring their weight when immersed in an inert liquid of known density. The weighing unit is a simple Jolly balance with a calibrated fine spiral steel spring whose sensitivity is about ±1.5 mg. The liquid is a Dow-Corning DC-200 silicone oil with a viscosity of about two centistokes at room temperature. This fluid does not swell the polyurethane resin-binder nor does it dissolve the ammonium perchlorate oxidizer, yet it is of sufficiently low viscosity to wet the specimens readily and completely. No appreciable penetration of the silicone fluid into propellants was observed during long periods of immersion; however, the catalyst ferric acetylacetonate is slightly soluble in the oil and is gradually extracted from immersed propellant samples. This results in a visually severe discoloration of the oil, but the quantity of dissolved catalyst is actually small. The silicone fluid is readily purified by vacuum distillation. Analysis of the residue confirmed the presence of ferric acetylacetonate, but disclosed no binder or oxidizer fragments or other impurities.

The container of silicone fluid is surrounded by a thermostatically controlled constant-temperature bath. During initial experiments, temperature was held within ±0.2°C, and the system was gradually improved to its present constancy of ±0.05°C. The test temperature is measured to the nearest ±0.005°C with a differential thermometer.

The propellant specimens are rings 1/4 in. wide with an inside diameter of 2.25 in. and an outside diameter of 2.5 in., as shown in Fig. 3. The rings are cut on a lathe from a propellant tube, which is prepared by casting and curing in a specially constructed mold. The casting of propellant tubes is somewhat painstaking and difficult, but can be accomplished successfully with most conventional polysulfide and polyurethane propellants. The dimensions of the specimens are determined accurately with a conical plug gage and a standard thickness gage (Fig. 3).

Figure 3 also shows some of the mandrels used to extend the propellant rings to strains ranging from 0.025 to 0.35, based on the inside diameter of the ring. It is true that the inside and outside of a ring are not stretched equally by this method, but the error is minimized by making the ratio of outside to inside diameter small (1.1/1). The upper section of each mandrel is conical to permit smooth and rapid extension of the propellant rings and the lower cylindrical section has a diameter calculated to give the desired strain. The aluminum mandrels are hollow, and their bulk density is adjusted to exceed slightly the density of the silicone oil at the test temperature. The weight and volume of each mandrel are accurately known. Each immersed mandrel puts only a slight load on the Jolly balance spring and permits high precision in determining the volume of the stretched propellant.

The apparatus and the experimental procedure were designed with the hope of being able to determine the Poisson's ratio not only as a function of strain but also as a function of time. To make a measurement, a propellant ring of known dimensions, weight, and volume is extended rapidly by sliding over the appropriate mandrel, as shown in one example in Fig. 3. The assembly is suspended from the Jolly balance spring in the silicone oil, and readings of apparent weight are taken as a function of time. From these data, changes in volume (if any) with time can be calculated for any one value of strain. After an equilibrium volume is reached, the ring is removed from the mandrel and the extent of volume recovery is measured.

Preliminary measurements were made with the three polyurethane propellants described in Table II. A more complete study, including an error analysis of data, is being carried out with two STARFINDER propellants, F-39GS and F-39SS (Table III). Samples of F-39GS, batch PF-218 were also sent to Aerojet-General Corp., Sacramento, for evaluation of Poisson's ratio by a dilatometer method which they are also developing (5). The STARFINDER propellants were prepared in the Planetex and Becken mixers, respectively; the slight difference in their composition is designed

to compensate for this difference in the mixing procedure and to give cured propellants with equivalent mechanical properties. Comparison of batches CL-33 and CL-34 bears this out; however, mechanical properties of subsequent batches of F-39GS propellant differed appreciably from the first one. The tensile properties at maximum load of batch PF-218 over a wide temperature range are shown in Fig. 4.

PRELIMINARY RESULTS

Data for the three polyurethane propellants of Table II are shown in Fig. 5, calculated as ν_1. They represent the maximum volume increase which was observed for any imposed strain, regardless of the time at which it occurred. The dotted line represents Poisson's ratio calculated from Equation 2 for an incompressible material, and it is seen that values for the three propellants decrease about twice as fast as the theoretical curve, and differ appreciably from each other. It should be noted, however, that the measurements with F-40BS propellant were made at 30° C, whereas the other two propellants were tested at 25° C. The existence of a critical strain ϵ_c is denied by these results, since measured values of the Poisson's ratio deviate from the theoretical curve even at the smallest strain. Thus, it seems that dewetting of oxidizer particles is a continuous process which begins as soon as strain is applied, in agreement with the Aerojet results (2).

When the data are plotted as V/V_0 vs $(1 + \epsilon_1)$, the results for X535 and F40-BS are linear above about 12% strain. Plots of log V/V_0 vs log $(1+\epsilon)$, to determine ν_3, show continuous curvature of all strains. The linear region in such plots found at high strains by Smith (3) for polyvinyl chloride-glass bead composites is never attained in the propellants, probably because of the relatively low breaking elongation.

The differential Hencky Poisson's ratio ν_3 for these and a STARFINDER propellant are shown in Fig. 6.

POISSON'S RATIO OF STARFINDER PROPELLANTS

Since the propellants tested had relatively poor elongation (Table III), data beyond 20% strain could not be obtained. Even at 20% strain, all rings broke after 30 to 50 min.

Results obtained at 25° are shown in Fig. 7. The points indicate the average values of Poisson's ratio at strains ranging from 2.5 to 20% at intervals of 2.5%, and the length of each vertical bar represents the variation of this value as a function of time over a 60-min period. The data are best represented by a straight line.

The results for propellant F-39GS batch PF-218 are also shown in Fig. 8, where they are compared with the Aerojet data[1] on the same propellant. Their results for two of the samples are in good agreement with each other and with the remainder of the data. The dotted line gives the expected error limit, as discussed below. Aerojet reported that they obtain better sample-to-sample reproducibility with their propellant than they found with the JPL samples. Apparently the samples were damaged in shipment, since we would also expect better reproducibility. Nevertheless, the two measurements gave substantially the same result for strains up to 15%. Moreover, both sets of experiments show that volume increases occur over the full range of strains.

ERROR ANALYSIS OF DATA

The variation of Poisson's ratio ν with time was found to be small and highly erratic. In many instances, little or no measurable change with time was found. Thus, it became important to examine the errors inherent in the experimental procedure and to determine the limits of reliability for the calculated values of ν. The method of analysis is described in most standard texts on statistics or error theory: when a dependent variable y is a function of k independent variables,

$$y = F(x_1, x_2, \cdots x_k), \tag{5}$$

then the variance in y is approximated by

$$\sigma_y^2 \cong \left(\frac{\delta F}{\delta x_1}\right)^2 \left(\sigma_{x_1}\right)^2 + \left(\frac{\delta F}{\delta x_2}\right)^2 \left(\sigma_{x_2}\right)^2 + \cdots \left(\frac{\delta F}{\delta x_k}\right)^2 \left(\sigma_{x_k}\right)^2 \tag{6}$$

Equations necessary to calculate ν from measured quantities and the corresponding expressions for σ are given in Table IV. The equation for σ_ν was simplified by validly assuming that the ratio V/V_0 is sufficiently close to unity to be omitted from error calculations, and also by the fact that σ_{V_0} equals σ_V. The input data and intermediate results are summarized in Table V. Maximum standard deviations of most independent variables were estimated on the basis of experience obtained during preliminary studies. This is perfectly adequate, because the calculation reveals that two of the measured quantities dominate in determining the standard deviation of ν. These are the spring constant of the Jolly balance K and the density of the silicone oil ρ. The error contribution of ρ was minimized by improving the temperature control and measurement, and by determining density accurately to five significant figures,[2] leaving K as the principal source of error

[1]We are indebted to Dr. Paul Colodny for these measurements.

[2]We are indebted to J. B. Krasinsky and S. P. Vango for these measurements.

in calculating ν. Consequently, σ_k was determined rigorously by following the procedure outlined in Ref. 3. The spring calibration was carried out over the full range of spring elongations as well as over the narrow ranges actually used in the experimental procedure, but the results were identical in both instances, giving $\sigma_k = 3.7 \times 10^{-3}$.

Final results of the error analysis are shown in Fig. 8, using the curve for STARFINDER propellant (Fig. 7). The outer solid envelope is calculated from initial estimated values of σ. The inner dashed envelope represents the minimum deviation attainable with the present apparatus. The actual experimental conditions were intermediate between these two extremes, because both the apparatus and the procedure were continually improved as the work progressed. The vertical bars represent all experimental data at any one strain, including variations as a function of time. Considering that the available sample is small (6), the agreement of experiment with the error analysis is good. The large experimental variance experienced at 20% strain may be due to the fact that the propellant was close to its breaking elongation.

It is clearly evident from Fig. 8 that the observed minute variations of Poisson's ratio with time are well within the limits of error, and consequently cannot be regarded as real. Moreover, even a significant reduction of σ_k would not improve the precision sufficiently to be able to decide whether or not the observed time dependence of ν is real and attributable to the sample. However, the time-independent values of Poisson's ratio as a function of strain can be measured satisfactorily, and for this purpose the present apparatus and method are superior to previously employed direct measurements of length and width.

The rate and extent of volume recovery after extension were also measured during a 60-min relaxation period. A residual increase in volume was generally found after strains greater than 5%. This "permanent" volume set ranged from 0.1 to 0.5% of the original specimen volume, or expressed in another way, from 13 to 45% of the maximum volume increase when strained. The data were not reproducible, however, and for the most part fell within the calculated limits of error. It can be concluded qualitatively that complete volume recovery does not take place in 60 min, but the data are not sufficiently precise for quantitative calculations.

CONCLUSIONS

An apparatus for measuring Poisson's ratio with some precision has been developed and tested. Analysis of the data shows that it gives reproducible results within experimental error. The latter is shown to be very large at small strains. A more sophisticated apparatus will be required to measure time dependent properties, should they remain as small as observed at room temperature.

This apparatus and that at Aerojet give comparable results and show that volume changes occur even at very low strains.

ACKNOWLEDGEMENT

Much of the experimental work was carried out by Mr. C. W. Weekley.

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TABLE I. POISSON'S RATIO AS A FUNCTION OF ϵ_1 AND V/V_0 FOR THREE DEFINITIONS

Definition	Poisson's Ratio, $\nu = f(\epsilon_1, V/V_0)$	
	No volume change, $V/V_0 = 1$	Volume change, $V/V_0 \neq 1$
Classical Cauchy $\nu_1 = -\frac{\epsilon_w}{\epsilon_1}$	$\nu_1 = \frac{1}{\epsilon_1}\left[1 - (1+\epsilon_1)^{-1/2}\right]$	$\nu_1 = \frac{1}{\epsilon}\left[1 - \left(\frac{V}{V_0}\right)^{1/2}(1+\epsilon_1)^{-1/2}\right]$
Differential Cauchy $\nu_2 = -\frac{d\epsilon_w}{d\epsilon_1}$	$\nu_2 = 1/2(1+\epsilon_1)^{-3/2}$	$\nu_2 = 1/2\left(\frac{V}{V_0}\right)^{1/2}(1+\epsilon_1)^{-3/2} - 1/2(1+\epsilon_1)^{-1/2}\left(\frac{V}{V_0}\right)^{-1/2}\left[\frac{d\left(\frac{V}{V_0}\right)}{d(1+\epsilon_1)}\right]$
Differential Hencky $\nu_3 = -\frac{d\ln(1+\epsilon_w)}{d\ln(1+\epsilon_1)}$	$\nu_3 = 1/2$	$\nu_3 = 1/2 - 1/2\left[\frac{d\ln\left(\frac{V}{V_0}\right)}{d\ln(1+\epsilon_1)}\right]$

TABLE II. COMPOSITION OF POLYURETHANE PROPELLANTS[a]

Composition, wt%	F-40BS HU-1	X-535 S-101	X-600 PD-79
Ammonium perchlorate, unground	53.78	56.00	55.87
Ammonium perchlorate, ground	23.05	24.00	23.94
Lithium perchlorate	-----	-----	0.19
Aluminum MD-105	2.00	-----	-----
Copper chromite Cu 0202	1.50	-----	-----
Polypropylene glycol 2025	16.68	17.20	13.31
P-II polymer[b]	-----	-----	5.03
1, 2, 3-trimethylol propane	0.20	0.09	0.09
Alrosperse 11p[c]	0.48	0.50	-----
Phenyl-β-naphtylamine	0.25	0.25	0.25
Ferric acetylacetonate	0.04	0.04	0.01
2, 4-toluene diisocyanate	2.02	1.92	1.31
Cure Conditions			
Cure temperature, °F	120	140	180[d]
Cure time, hours	72	72	88[d]
Measured Initial Mechanical Properties at 77°F[e]			
Tensile strength, psi	-----	112.3	152.2[d]
Elongation at maximum load, %	-----	91.8	40.6[d]

[a]Column headings indicate propellant type and batch number.
[b]Polymer prepared from 2 moles of polypropylene glycol, 4 moles of 2, 4-toluene diisocyanate, and 1 mole of 2, 4-toluene diamine.
[c]Wetting agent by Geigy Industrial Chemicals.
[d]Undercured under these conditions; completion of cure by holding at 120°F for 2 weeks gives a tensile strength of 180 to 200 psi and an elongation at maximum load of about 35%.
[e]Approximate strain rate 0.74 min^{-1}.

TABLE III. COMPOSITION AND MECHANICAL PROPERTIES OF STARFINDER PROPELLANTS

Composition, wt%	F-39GS	F-39SS
Ammonium perchlorate, unground	53.62	53.62
Ammonium perchlorate, ground	22.98	22.98
Aluminum MD-105	2.00	2.00
Lithium fluoride	1.75	1.75
Polypropylene glycol 2025	16.88	16.82
1, 2, 3-trimethylol propane	0.09	0.10
Alrosperse 11p[a]	0.48	0.49
Phenyl-β-naphthylamine	0.25	0.25
Ferric acetylacetonate	0.04	0.04
2, 4-toluene diisocyanate	1.91	1.95
Cure Conditions		
Cure temperature, °F	120	120
Cure time, hours	120	120

Measured Initial Mechanical Properties	F-39GS CL-33	F-39SS CL-34	F-39GS PF-217	F-39GS PF-218
Tensile strength, psi	147.3[b]	138.0[b]	127.1[c]	133.5[c]
Elongation at maximum load, %	22.4[b]	21.9[b]	28.2[c]	32.2[c]

[a]Wetting agent by Geigy Industrial Chemicals.
[b]80°F; nominal strain rate 0.37 min^{-1}.
[c]77°F; nominal strain rate 0.74 min^{-1}.

TABLE IV. EQUATIONS FOR CALCULATING DEPENDENT VARIABLES AND THEIR STANDARD DEVIATIONS
(Symbols are identified in Table V)

Dependent Variables	Standard Deviations of Dependent variables
$W_I = \frac{R_f - R_0}{K}$	$\sigma_{W_I}^2 = \left(\frac{1}{K}\sigma_{R_F}\right)^2 + \left(\frac{1}{K}\sigma_{R_0}\right)^2 + \left(\frac{R_F - R_0}{K^2}\sigma_K\right)^2$
$V = \frac{W_a - W_I}{\rho}$	$\sigma_V^2 = \left(\frac{1}{\rho}\sigma_{W_A}\right)^2 + \left(\frac{1}{\rho}\sigma_{W_I}\right)^2 + \left(\frac{W_A - W_I}{\rho^2}\sigma_\rho\right)^2$
$\alpha = \frac{L}{L_0}$	$\sigma_\alpha^2 = \left(\frac{1}{L_0}\sigma_L\right)^2 + \left(\frac{L}{L_0^2}\sigma_{L_0}\right)^2$
$\nu = \frac{1}{\alpha - 1}\left[1 - \left(\frac{V}{V_0}\right)^{1/2}\alpha^{-1/2}\right]$	$\sigma_\nu^2 = \left[(1/2)\frac{\alpha^{3/2}}{\alpha - 1} - \frac{1}{(\alpha - 1)^2}(1 - \alpha^{-1/2})\right]^2 \sigma_\alpha^2 + 2\left[1/2\,\frac{1}{V_0}\frac{\alpha^{-1/2}}{\alpha - 1}\right]^2 \sigma_V^2$

TABLE V. INPUT DATA FOR ERROR ANALYSIS OF POISSON'S RATIO DETERMINATIONS

Measured (Independent) Variables	Symbol	Units	Magnitude	Maximum Standard Deviation[a]	Minimum Standard Deviation[b]
Initial reading of Jolly balance	R_0	cm	$R_f - R_0 = 20$	5×10^{-3}	5×10^{-3}
Final reading of Jolly balance	R_f	cm		5×10^{-3}	5×10^{-3}
Spring constant of Jolly balance	K	cm/g	6.75	1×10^{-2}	3.7×10^{-3}
Temperature	t	°C	25.0	2×10^{-1}	5×10^{-2}
Density of silicone oil	ρ	g/cc	0.87	1×10^{-3}	5×10^{-5}
Weight of sample in air	W_A	g	6.5	5×10^{-4}	5×10^{-4}
Inside circumference of ring	L_0	cm	2.25π	1×10^{-3}	1×10^{-3}
Outside circumference of mandrel	L	cm	$2.307\pi - 3.040\pi$	1×10^{-3}	1×10^{-3}
Calculated (Dependent) Variables					
Weight of sample in oil	W_I	g	3.0	4.5×10^{-3}	1.9×10^{-3}
Initial volume of sample	V_0	cc	3.8	7×10^{-3}	2.3×10^{-3}
Volume of extended sample	V	cc	3.8	7×10^{-3}	2.3×10^{-3}
Extension ratio	α	-	1.025 - 1.350	6.8×10^{-4}[c]	6.8×10^{-4}[c]
Poisson's ratio	ν	-	see Fig. 8	see Fig. 8	

[a]Maximum observed during preliminary measurements.

[b]Best possible with currently improved apparatus.

[c]Actually varies slightly from 6.4×10^{-4} to 7.5×10^{-4}.

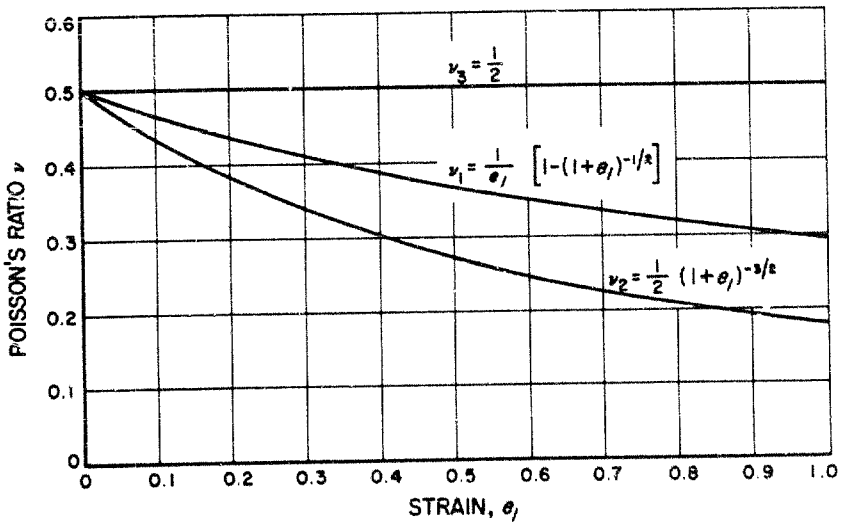

FIGURE 1. POISSON'S RATIO FOR INCOMPRESSIBLE MATERIALS AS A FUNCTION OF ϵ_1 FOR THREE DEFINITIONS (SEE TABLE I)

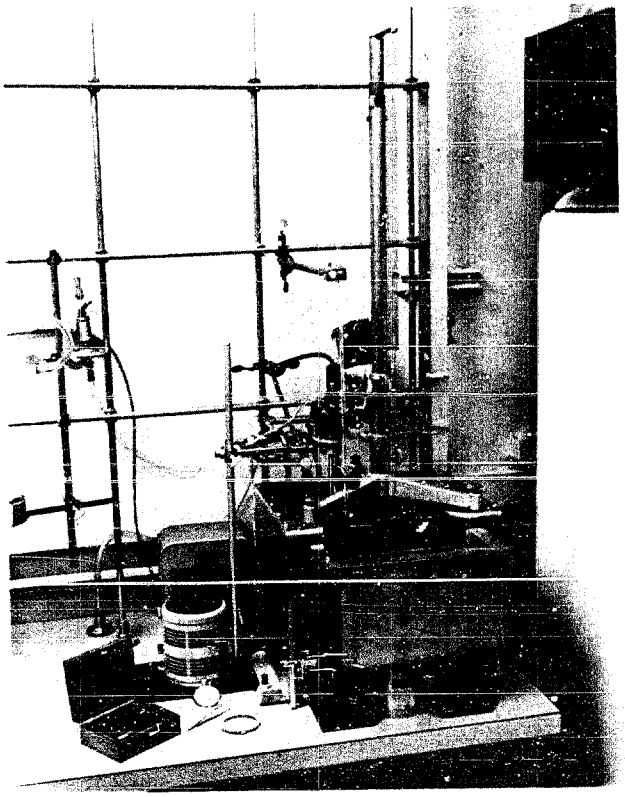

FIGURE 2. VOLUME-CHANGE TEST APPARATUS

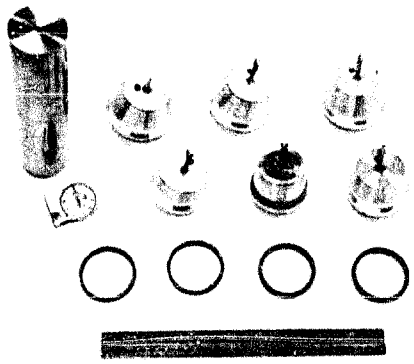

FIGURE 3. VOLUME-CHANGE TEST SPECIMENS AND STRAIN MANDRELS

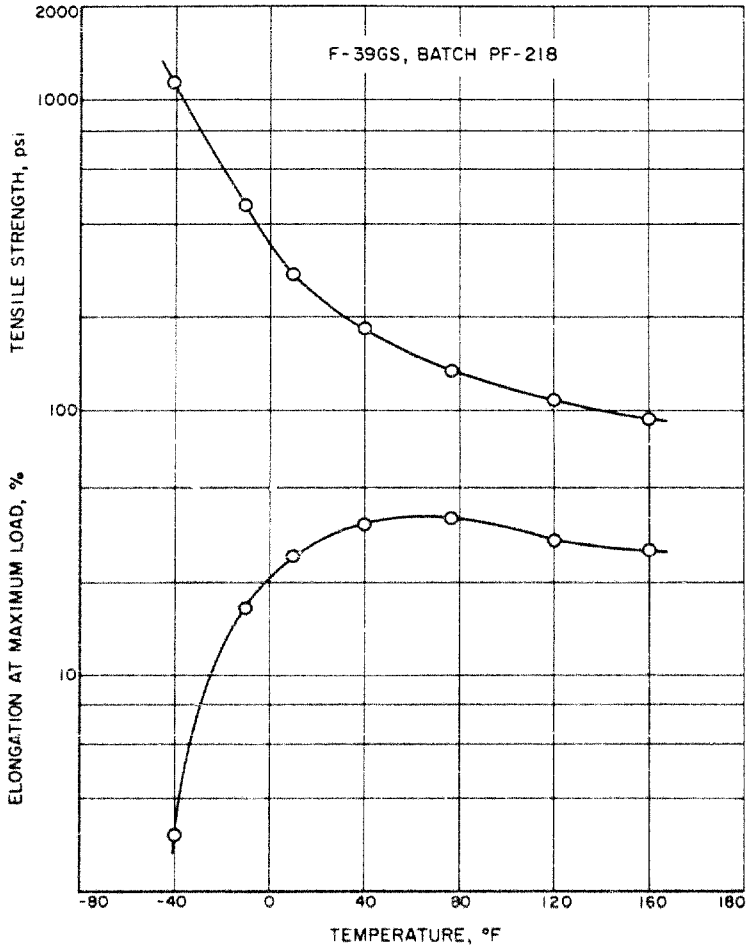

FIGURE 4. MECHANICAL PROPERTIES OF STAR-FINDER PROPELLANTS

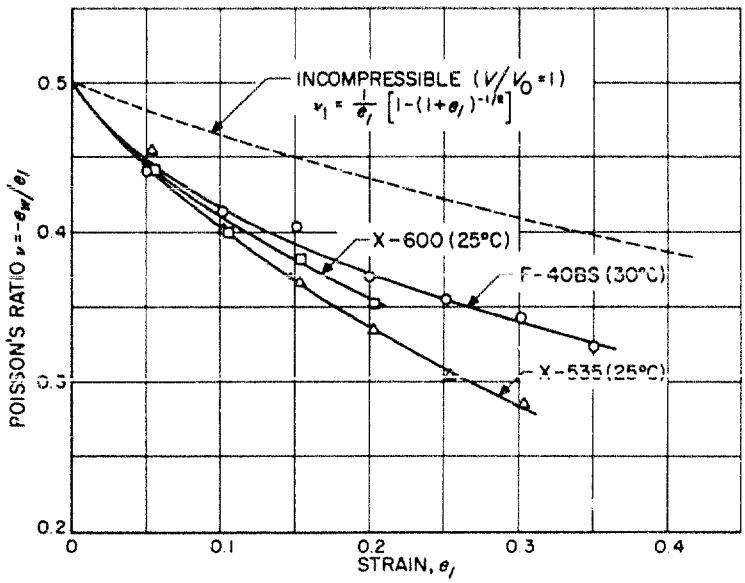

FIGURE 5. POISSON'S RATIO AS A FUNCTION OF STRAIN FOR THREE POLYURETHANE PROPELLANTS

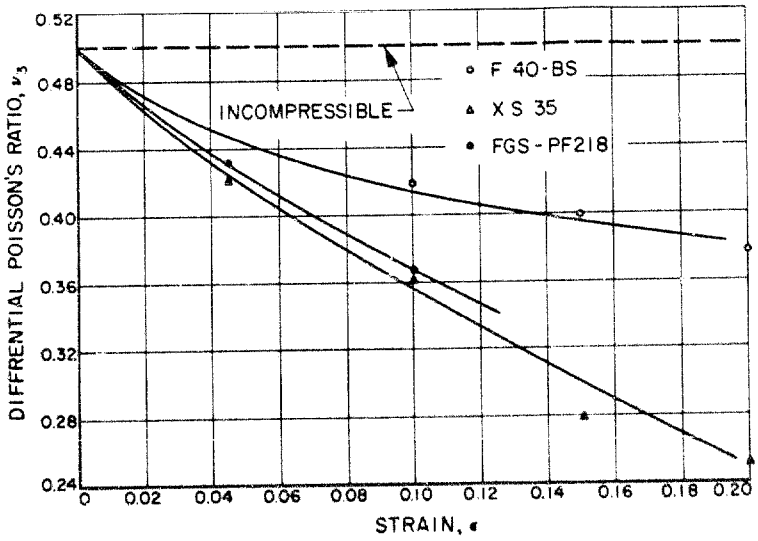

FIGURE 6. DIFFERENTIAL POISSON'S RATIO ν_3 FOR 3 POLYURETHANE PROPELLANTS

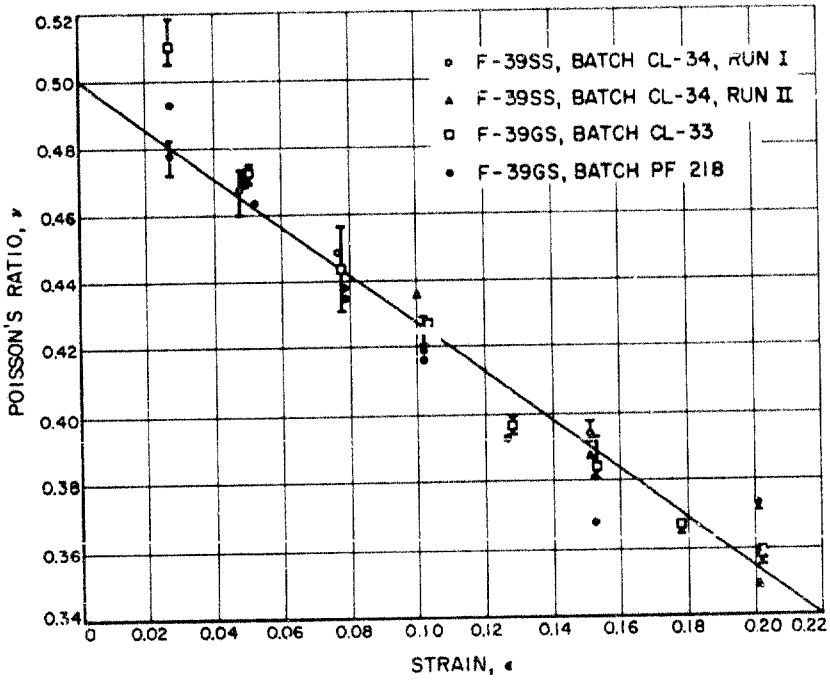

FIGURE 7. POISSON'S RATIO AS A FUNCTION OF STRAIN FOR STARFINDER PROPELLANTS

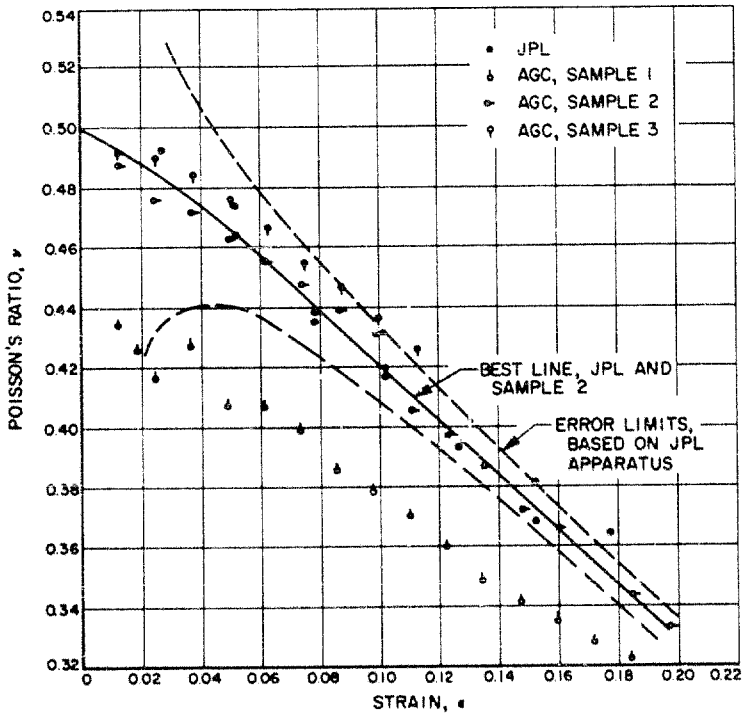

FIGURE 8. COMPARISON OF POISSON'S RATIO AS DETERMINED AT JPL AND AT THE AEROJET GENERAL CORP., PROPELLANT F39GS/PF218

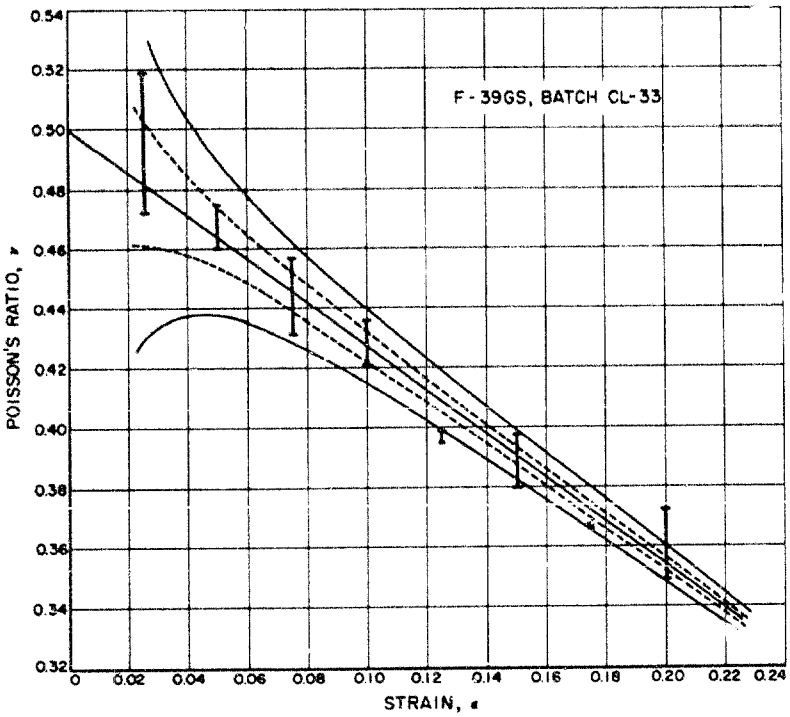

FIGURE 9. ERROR ANALYSIS OF POISSON'S RATIO DETERMINATIONS

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